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A new method for the analysis of compaction processes in high-porosity agglomerates

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Abstract Mechanical properties of high-porous microscopic agglomerates have been investigated. For this purpose we installed an atomic force microscope (AFM) cantilever in a scanning electron microscope (SEM) using a nanomanipulator. The nanomanipulator is piezoelectric controlled with increments of 5 nm in the rotational and 0.5 nm in the translational direction. Thus, this tool allows the precise positioning and movement of an AFM cantilever under SEM observation. Depending on the spring constant of the cantilever and the step size of the motion—both quantities determining the sensitivity of the instrument—different aspects of the deformation of dust-aggregate structures, e.g., the behaviour of single particle chains, can be analysed.

Keywords Nanomanipulator · SEM · AFM · High-porous agglomerate

1 Introduction

Granular matter and its properties is a big issue in fundamental research as well as in engineering. Taking into account that granular matter is the second most handled material by men—after water—the interest in a deeper understanding is self-evident. One example in fundamental research where the interaction of micron sized particles as well as properties of agglomerates consisting of these particles is of importance is the formation of planetesimals, the kilometre-sized precursors of the solid planetary bodies in our solar system [1]. Some phases of the planet-formation process are dominated by collisions between and impacts of high-porosity dust aggregates. The understanding of the physical properties of such structures is of relevance for the whole planetesimal-formation process, spanning nine orders of magnitude in size and 27 orders of magnitude in mass.

Beside the information that can be derived directly from the controlled compaction of dust aggregates with both, precise knowledge of the applied force as well as high spatial resolution of the structure's response, a comparison with the results of computer simulations, offers the chance to validate and improve the simulation models. The comparison between discrete element simulations [2] and macroscopic experiments suffer from the difference in the particle numbers involved. Those numbers differ by orders of magnitude. With our microscopic experiments, this discrepancy can be overcome. Hit-and-stick deposition processes in both cases (the experimental and the numerical), leading to structures with the same porosity and comparable particle number, offer the chance to compare models with reality, leading to a better understanding of the compaction process. Beside the force–displacement comparison in both compaction processes, this scanning electron microscopy (SEM) controlled compaction offers a chance to follow and analyse individual particle trajectories that can also be compared with the same aspect in the simulation, where all individual particle reactions are given.

To establish a model relating the bulk's porosity to the compaction stress for the beginning compaction, using physically relevant parameters, investigations like these might be a first step.

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2 Experimental

Two experimental procedures are required to investigate the compaction of high-porosity microscopic dust agglomerates. First, the structures that shall be compacted have to be generated and second, the compaction has to be controlled concerning two aspects: the applied force and the local displacement induced.

For the formation of microscopic dust aggregates, we initiated a process referred to as random ballistic deposition (RBD) [3]. A powder sample is deagglomerated into its monomer grains by the use of a cogwheel [4] and accelerated into rarefied air with a typical pressure of 100 Pa where they couple to a laminar gas flow. The cohesion of the particles due to van der Waals forces, about 60 nN [5] in the case of 1.5 μm SiO_2 particles, is more than six orders of magnitude higher than the particle weight. The particles' ballistic trajectories in the laminar gas flow end in a hit-and-stick process on a solid target which leads to agglomerates with high porosities of about $p = 0.85$ [6]. Here, the porosity is defined through the mass density of the agglomerates, ρ_a, and the mass density of the monomer particles, ρ_p, by $p = 1 - \rho_a/\rho_p$. This growth process can be initiated on different target surfaces, i.e., either on filters, leading to macroscopic dust agglomerates [6], or on small structures to generate microscopic dust agglomerates. To investigate structures with a very small number of constituents, these microscopic dust aggregates can be used as precursors and tailored by the use of the nanomanipulator under SEM observation.

The second experimental procedure uses a nanomanipulator (Kleindiek, Reutlingen, Germany) that allows performing defined motions inside an SEM. The motions are caused by the extension or contraction of a piezo crystal under electric voltage. The piezo-driven probe allows movements of the tip in 5 nm increments in the rotational and 0.5 nm in the translational direction (see Fig. 1). Using a low current SEM (Leo 1530 with Gemini column) enables the investigation of pristine non-conducting dust agglomerates without any sample conditioning, such as the deposition of a conductive gold or carbon layer on the particles which would spoil the mechanical measurements irretrievably. The mounting of the nanomanipulator is equipped with an AFM cantilever (here: rectangular silicon cantilever, length 350 μm, width 35 μm, thickness 2 μm; NSC 12/tipless/without Al/50 from MikroMasch). The commonly used laser light lever technique to detect the deflection of the cantilever in AFM's can be omitted in this case, because the position of the cantilever's tip is detected by instantaneous SEM imaging. So the cantilever's deflection is ascertainable by the knowledge of the nanomanipulator motion.

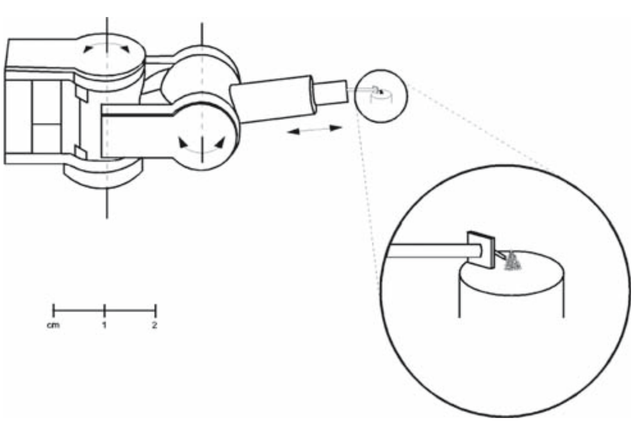

Fig. 1 Charting of the nanomanipulator, *arrows* showing the directions of movement related to the axes of motion. The *zoomed region* sketches the AFM cantilever as well as an agglomerate on the SEM sample holder. Linear measure is given in the figure

3 Results and discussion

In our experiments, we used monodisperse spherical SiO_2 particles (sicastar from micromod Partikeltechnologie GmbH, Rostock, Germany) with 1.5 μm diameter. The dust agglomerates were formed by the RBD technique (see Sect. 2) on a solid substrate. Moving the AFM cantilever by means of the nanomanipulator, the dust aggregates can be compacted (see Fig. 2).

The applied force can be calculated by multiplying the cantilever's spring constant with its deflection. For this transformation, the spring constant of the cantilever is essential. We determined the spring constant by the method of measuring a force–distance-curve with the unknown cantilever against a reference with known spring constant as described previously [7]. The spring constant of the reference cantilever was determined by analyzing the resonance frequency of the cantilever with the Sader method [8].

Analyzing the acting force while compacting the dust agglomerate by moving the nanomanipulator results in a force–distance-diagram (see Fig. 3).

This graph in Fig. 3 indicates the existence of substructures in the compaction process of dust agglomerates. The saw-tooth-like pattern of the force–distance-diagram is typical for microscopic compaction in contrast to macroscopic experiments. Beside the increase of the force with advancing compression, there are several peaks identifiable. The development of those peaks can be assigned to the movement of substructures visible in the SEM image sequence of the compaction process. A certain force value has to be reached before the structure reacts with motion of at least three individual particle–particle contacts. Before reaching this point, the force applied by the cantilever is countered by force chains in the agglomerate. The linear increase of the force with distance, represented by the dotted lines in Fig. 3, represents the movement of the cantilever with the motion of the nanomanipulator, the slope equals the spring constant

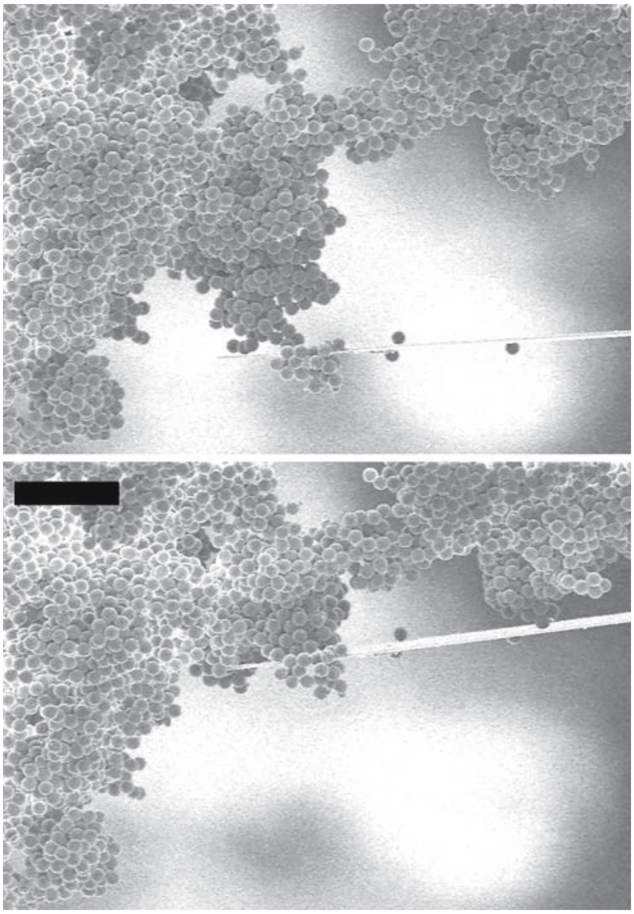

Fig. 2 Two SEM-images taken from the image sequence showing the whole compaction process. The images were taken after 10 and 34 μm of cantilever movement, indicated by the *stars* in Fig. 3. The *bar* indicates a length of 10 μm

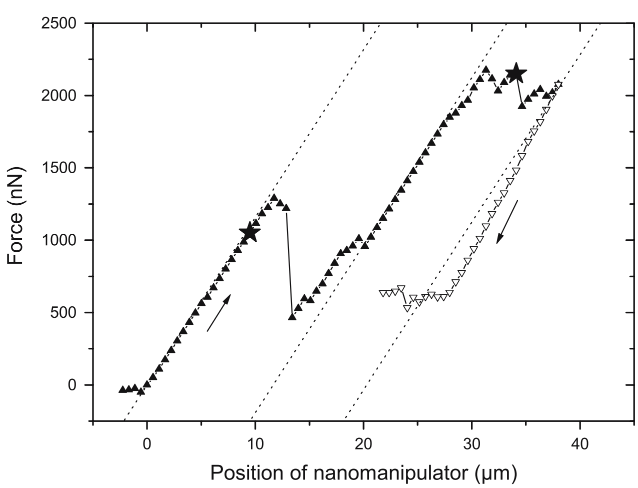

Fig. 3 The *diagram* shows the measured force applied by the cantilever to the dust agglomerate as a function of the traveled distance induced by the nanomanipulator

of the cantilever. In the retracting part of the curve in Fig. 3 (open symbols), the elasticity of the structure is visible due to the steeper slope of the curve.

4 Conclusions

We presented experiments of compacting high-porosity microscopic agglomerates under SEM control with the colloidal probe technique. This confirms the feasibility of the experimental approach to minimize the number of particles in the manipulated dust aggregate in order to (1) compare the results to computer simulations done with a three-dimensional contact dynamics method [9] and to (2) relate the mechanical properties of dust aggregates to single-particle properties, such as adhesion and friction forces. The spatial resolution in our experiments was 59.9 nm per pixel resulting in a force resolution of 6.9 nN. In principle multiplex aspects of particle particle interactions can be focused on, depending on the spring constant of the applied cantilever, the chosen step size of the nanomanipulator movement and the magnification of the imaging, the sensitivity can be tuned.

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